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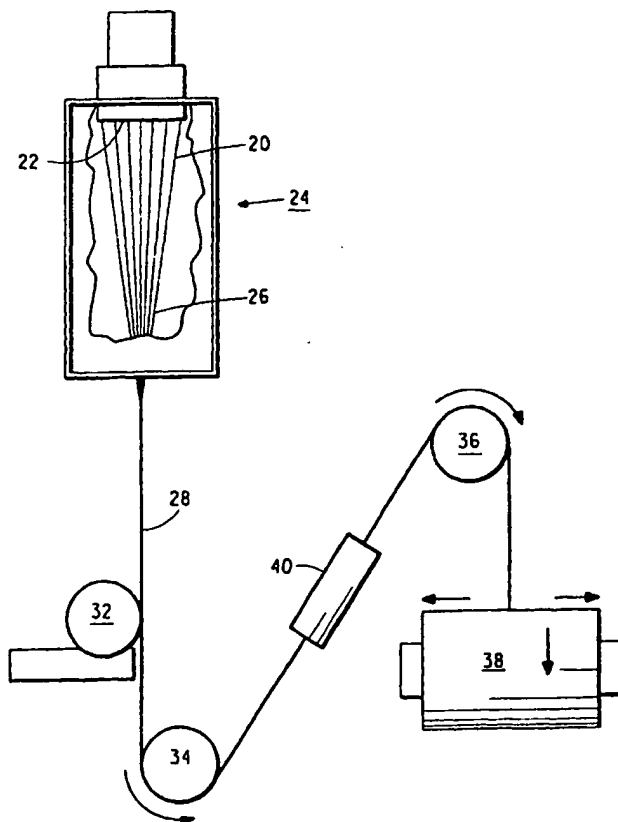
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(54) Title: **FINE DENIER YARN FROM POLY(TRIMETHYLENE TEREPHTHALATE)**



(57) Abstract: The invention is directed to fine denier poly(trimethylene terephthalate) feed yarns and drawn yarns and their manufacture. The yarns are drawn such that the actual draw ratio is within 10 percent of the predicted draw ratio determined according to:  $\frac{[(\text{elongation to break of the feed yarn}) + 115]}{[(\text{elongation to break of the drawn yarn}) + 115]}$ .

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*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

**TITLE OF INVENTION****FINE DENIER YARN FROM POLY(TRIMETHYLENE  
TEREPHTHALATE)**

5

**FIELD OF THE INVENTION**

The present invention relates to very fine denier polyester yarn made from poly(trimethylene terephthalate) fibers.

**BACKGROUND OF THE INVENTION**

10 Polyester yarns having very fine denier are highly desirable for manufacturing fabrics used in the garment industry. Such yarns are desirable because they yield a light-weight material having excellent properties such as softness. The softness of a yarn and fabric is a measure of how soft a material feels to the touch. A yarn and fabric used for many clothing apparel items require  
15 a high degree of softness.

Very fine denier polyester fibers currently known in the art are made using polyethylene terephthalate. Such yarns provide softness suitable for many garments such as, e.g., dresses, jackets and other ladies' apparel. However, because polyethylene terephthalate has a high Young's modulus, the maximum  
20 softness achieved is not suitable for garments requiring ultra-soft touch.

There is therefore a need in the art for very fine denier polyester yarns having superior softness quality. Theoretically, polyester yarns made from a polymer having a low Young's modulus should yield the desirable properties. However, attempts to commercially manufacture such a fine denier polyester yarn  
25 from poly(trimethylene terephthalate) have not been successful due to various manufacturing problems. For example, when attempting to make very fine denier yarns from poly(trimethylene terephthalate), excessive breaks in the fibers have been experienced. Further, it was thought in the prior art that the tenacity of poly(trimethylene terephthalate) was too low to successfully make a very fine  
30 denier yarn.

**SUMMARY OF THE INVENTION**

This invention is directed to a process for making a drawn yarn comprising: (a) providing partially oriented feed yarn filaments prepared from a polyester polymer having an intrinsic viscosity of at least 0.80 dl/g comprising at  
35 least 85 mole % poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units; and (b) drawing the filaments between a set of feed rolls to produce a denier per filament less than about 1.5 and an actual draw ratio within 10 percent of a predicted draw ratio, wherein the predicted draw ratio is determined according to: [(elongation to break of the feed

yarn) + 115]/[(elongation to break of the drawn yarn) + 115)]. Preferably the process further comprises heating the filaments to a temperature greater than the glass transition temperature of the filaments, but less than 200°C, prior to drawing the filaments.

- 5            Preferably process further comprises preparing the partially oriented feed yarn filaments by extruding the polyester in a molten state a temperature between about 255°C and 275°C through a spinneret to form filaments.

            In one embodiment, the process also comprises interlacing the filaments prior to drawing them.

- 10           Preferably the actual draw ratio is within 5 percent of the predicted draw ratio, more preferably within 3 percent of the predicted draw ratio.

            Preferably the denier per filament of the drawn yarn is less than 1.0.

- Preferably the undrawn filaments have a denier per filament less than about 2, more preferably less than about 1.0. By "undrawn" reference is made to  
15 the filaments prior to carrying out the drawing step, and the skilled artisan will recognize that these filaments are partially drawn in preparing the partially oriented yarn.

- The invention is also directed to the process wherein the drawing comprises warp drawing or single end drawing and further comprising air jet  
20 texturing or false-twisting.

- The invention is further directed to a process of preparing a fine denier partially oriented undrawn feed yarn made from a polyester polymer melt-extruded at a spinning temperature between about 255°C and about 275°C, wherein said polymer comprises at least 85 mole % poly(trimethylene  
25 terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units, and wherein said polymer has an intrinsic viscosity of at least 0.80 dl/g, and wherein said partially oriented intrinsic viscosity of at least 0.80 dl/g, and wherein said partially oriented undrawn fine denier feed yarn has a denier per filament less than about 2.

- 30           The process of claims 8 or 10 wherein the undrawn filaments have a denier per filament less than about 1.5.

            Preferably the denier per filament of the drawn yarn is less than 1.0.

            Preferably the undrawn filaments have a denier per filament less than about 2, more preferably less than 1.5 and most preferably less than 1.0.

- 35           Preferably, the polymer has an intrinsic viscosity of 0.90 dl/g, more preferably 1.00 dl/g.

            Preferably, the spinning temperature is between 260°C and 270°C.

Preferably, the polyester is melt-extruded on a spinneret having orifices between about 0.12 to 0.38 mm in diameter.

The invention is also directed to a yarn prepared by the process of any of the preceding claims.

5       The invention is further directed to a drawn yarn prepared from a polyester polymer having an intrinsic viscosity of at least 0.80 dl/g comprising at least 85 mole % poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units, wherein the drawn yarn has a denier per filament less than about 1.0.

10       The invention is also directed to a drawn yarn made by the process of: (1) providing filaments of a partially oriented feed yarn spun from a polyester polymer, preferably prepared by melt-extruding the polyester polymer at a temperature between about 255°C and 275°C, wherein the polyester polymer has an intrinsic viscosity of at least 0.80 dl/g and comprises at least 85 mole %  
15 poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units; and (2) preparing a drawn yarn from the partially oriented feed yarn, wherein said drawn yarn has the following characteristics: (a) a denier per filament less than about 1.0; and (b) an actual draw ratio within 10 percent of a predicted draw ratio, wherein the predicted draw ratio is determined  
20 according to:  $[(\text{elongation to break of the feed yarn}) + 115]/[(\text{elongation to break of the drawn yarn}) + 115]$ .

In addition, the invention is directed to a drawn yarn made by the following process: (1) providing a polyester polymer having an intrinsic viscosity of at least 0.80 dl/g comprising at least 85 mole % poly(trimethylene  
25 terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units; (2) spinning the polyester polymer by melt-extruding the polyester polymer at a temperature between about 255°C and 275°C to form a partially oriented feed yarn; (3) preparing a drawn yarn from the partially oriented feed yarn, wherein said drawn yarn has the following characteristics: (a)  
30 a denier per filament less than about 1.0; and (b) an actual draw ratio within 10 percent of a predicted draw ratio, wherein the predicted draw ratio is determined according to:  $[(\text{elongation to break of the feed yarn}) + 115]/[(\text{elongation to break of the drawn yarn}) + 115]$ .

The present invention also comprises a drawn yarn made from a partially  
35 oriented feed yarn, said feed yarn made from a polyester polymer melt-extruded at a spinning temperature between about 255°C and 275°C, wherein said polymer comprises at least 85 mole % poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units, and wherein said polymer

has an intrinsic viscosity of at least 0.80 dl/g, and wherein said drawn yarn has the following characteristics: (a) a denier per filament less than about 1.5; and (b)

an actual draw ratio within 10 percent of a predicted draw ratio, wherein the predicted draw ratio is determined according to:  $[(\text{elongation to break of the feed yarn}) + 115]/[(\text{elongation to break of the drawn yarn}) + 115]$ , and the process of making such a drawn yarn.

The present inventions further comprises a fine denier feed yarn made from a polyester polymer melt-extruded at a spinning temperature between about 255°C and about 275°C, wherein said polymer comprises at least 85 mole % poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units, and wherein said polymer has an intrinsic viscosity of at least 0.80 dl/g, and wherein said fine denier feed yarn has a denier per filament less than about 2.

### DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic diagram of an exemplary spinning position for making the very fine denier poly(trimethylene terephthalate) yarns of the present invention.

### DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a very fine denier polyester drawn yarn made from poly(trimethylene terephthalate) and a feed yarn and process for making the same. The very fine denier feed yarn of the present invention is a multifilament yarn wherein the denier per filament is less than about 2 dpf (2.22 dtex/filament). Preferably, the denier per filament of the feed yarn is less than 1.5 dpf (1.67 dtex/filament) and, most preferably, the denier per filament is less than 1 dpf (1.11 dtex/filament). The feed yarn denier per filament can be as low as 0.75, or even smaller. The very fine denier drawn yarn of the present invention is a multifilament yarn wherein the denier per filament is less than about 1.5 dpf (1.67 dtex/filament). Preferably, the denier per filament is less than 1 dpf (1.11 dtex/filament). The very fine denier drawn yarn can have a denier per filament of 0.65 dpf, preferably as low as 0.5 dpf, or smaller. The feed yarns (and consequently, the drawn yarns) are made from a polyester polymer, wherein said polymer comprises at least 85 mole % poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units, and wherein said polymer has an intrinsic viscosity of at least 0.80 dl/g. Preferably, the intrinsic viscosity is at least 0.90 dl/g and, most preferably, it is at least 1.00 dl/g. Preferably, the polymer has an intrinsic viscosity of 1.5 dl/g or less, more preferably 1.2 dl/g or less. Partially oriented feed yarn is made using conventional melt-spinning techniques, at a spinning temperature of about 255°C

to about 275°C. Molten polymer is extruded through spinneret orifices of diameter from about 0.12 mm to about 0.38 mm. The yarns of the present invention are drawn such that actual draw ratio is within ten percent of the predicted draw ratio. This requirement is satisfied if the draw ratio difference,  $\Delta DR$ , is less than ten percent. The draw ratio difference,  $\Delta DR$ , as defined herein is defined according to equation (I):

$$(I) \Delta DR = \left| \frac{DR_P - DR_A}{DR_A} \right| \leq 10\%,$$

where  $DR_A$  is the actual draw ratio, and  $DR_P$  is the predicted draw ratio. The predicted draw ratio,  $DR_P$  is defined according to equation (II):

$$(II) DR_P = \frac{E_B(F_Y) + 115}{E_B(D_Y) + 115},$$

where,  $E_B(F_Y)$  is the elongation to break of the partially oriented feed yarn and  $E_B(D_Y)$  is the elongation to break of the drawn yarn. Preferably, the actual draw ratio is within five percent of the predicted draw ratio and, most preferably, it is within three percent.

As shown in Figure 1, molten streams 20 of poly(trimethylene terephthalate) polymer are extruded through orifices in spinneret 22 downwardly into quench zone 24 supplied with radially or transversely directed quenching air. The diameter and quantity of orifices in spinneret 22 may be varied depending upon the desired filament size and the number of filaments in the multifilament yarn of the present invention. Further, the temperature of molten streams 20 is controlled by the spin block temperature, which is also known as the spinning temperature. It has been found that an orifice diameter of about 0.12 mm to about 0.38 mm can be used to produce the very fine filament yarns of the present invention. Further, a spinning temperature between about 255°C and 275°C is required to make the very fine denier yarns of the present invention. Preferably, the spinning temperature is between about 260°C and 270°C and, most preferably, the spinning temperature is maintained at 265°C.

Streams 20 solidify into filaments 26 at some distance below the spinneret within the quench zone. Filaments 26 are converged to form multifilament yarn 28. A conventional spin-finish is applied to yarn 28 through a metered application or by a roll application such as finish roll 32. Yarn 28 next passes in partial wraps about godets 34 and 36 and is wound on package 38. The filaments may be interlaced if desired, as by pneumatic tangle chamber 40.

The partially oriented poly(trimethylene terephthalate) yarns are then drawn using conventional drawing equipment, such as a Barmag DW48. According to the present invention, the yarns are drawn such that the draw ratio difference,  $\Delta DR$ , is less than ten percent, as described above.

5        The drawing can comprise warp drawing or single end drawing. The very fine filament yarns of the present invention are suitable for air jet texturing, false-twist texturing, gear crimping, and stuffer-box crimping, for example. The yarns of the present invention may be used to make any fabrics which could be made from very fine denier polyethylene terephthalate yarns, such as disclosed in U.S.  
10 Patent 5,250,245, which is incorporated herein by reference in its entirety. Tows made from these filament may also be crimped, if desired, and cut into staple and flock. The fabrics made from these improved yarns may be surface treated by conventional sanding and brushing to give suede-like tactility. The filament surface frictional characteristics may be changed by selection of cross-section,  
15 delusterant, and through such treatments as alkali-etching. The improved combination of filament strength and uniformity makes these filaments especially suited for end-use processes that require fine filament yarns without broken filaments (and yarn breakage) and uniform dyeing with critical dyes.

20        The fine filament yarns of the present invention are especially suitable for making high-end density moisture-barrier fabrics, such as rainwear and medical garments. The surface of the knit and woven fabrics can be napped (brushed or sanded). To reduce the denier even further, the filaments may be treated (preferably in fabric form) with conventional alkali procedures. The fine filament yarns of the present invention may be co-mingled on-line in spinning or off-line  
25 with higher denier polyester (or nylon) filaments to provide for cross-dyed effects and/or mixed shrinkage post-bulkable potential, where the bulk may be developed off-line, such as over feeding in the presence of heat while beaming/slashing or in fabric form, such as in the dye bath. The degree of interlace is selected based on the textile processing needs and final desired yarn/fabric aesthetics. Because of  
30 the low Young's modulus of poly(trimethylene terephthalate), the very fine denier yarns of the present invention are especially suitable for fabrics where softness is important.

      The fibers of this invention can have round, oval, octa-lobal, tri-lobal, scalloped oval, and other shapes, with round being most common.

35        Measurements discussed herein were made using conventional U.S. textile units, including denier, which is a metric unit. The dtex equivalents for denier are provided in parentheses after the actual measured values. Similarly, tenacity and



modulus measurements were measured and reported in grams per denier("gpd") with the equivalent dN/tex value in parentheses.

### **TEST METHODS**

5 The physical properties of the partially oriented poly(trimethylene terephthalate) yarns reported in the following examples were measured using an Instron Corp. tensile tester, model no. 1122. More specifically, elongation to break,  $E_B$ , and tenacity were measured according to ASTM D-2256.

Boil off shrinkage ("BOS") was determined according to ASTM D 2259 as follows: a weight was suspended from a length of yarn to produce a 0.2 g/d (0.18 dN/tex) load on the yarn and measuring its length,  $L_1$ . The weight was then removed and the yarn was immersed in boiling water for 30 minutes. The yarn was then removed from the boiling water, centrifuged for about a minute and allowed to cool for about 5 minutes. The cooled yarn is then loaded with the same weight as before. The new length of the yarn,  $L_2$ , was recorded. The percent shrinkage was then calculated according to equation (III), below:.

$$(III) \quad \text{Shrinkage (\%)} = \frac{L_1 - L_2}{L_1} \times 100$$

Dry heat shrinkage ("DHS") was determined according to ASTM D 2259 substantially as described above for BOS.  $L_1$  was measured as described, however, instead of being immersed in boiling water, the yarn was placed in an oven at about 160°C. After about 30 minutes, the yarn was removed from the oven and allowed to cool for about 15 minutes before  $L_2$  was measured. The percent shrinkage was then calculated according to equation (III), above.

Intrinsic viscosity was measured in 50/50 weight percent methylene chloride/trifluoroacetic acid following ASTM D 4603-96.

### **Example I – Polymer Preparation**

#### **Polymer Preparation 1**

Poly(trimethylene terephthalate) polymer was prepared using batch processing from dimethylterephthalate and 1,3-propanediol. A 40 lb (18 kg) horizontal autoclave with an agitator, vacuum jets and a monomer distillation still located above the clave portion of the autoclave was used. The monomer still was charged with 40 lb (18 kg) of dimethyl terephthalate and 33 lb (15 kg) of 1,3-propanediol. Sufficient lanthanum acetate catalyst was added to obtain 250 parts per million ("ppm") lanthanum in the polymer. Parts per million is used herein to mean micrograms per gram. In addition, tetraisopropyl titanate polymerization catalyst was added to the monomer to obtain 30 ppm titanium in the polymer. The temperature of the still was gradually raised to 245°C and approximately 13.5 lb (6.2 kg) of methanol distillate were recovered.

An amount of phosphoric acid in 1,3-propanediol solution to obtain about 160 ppm phosphorous in the polymer was added to the clave. If delustered polymer was desired, then a 20 percent by weight ("wt. %") slurry of titanium dioxide ( $\text{TiO}_2$ ) in 1,3-propanediol solution was added to the clave in an amount to  
5 give 0.3 wt. % in polymer. The ingredients were agitated and well mixed and polymerized by increasing the temperature to  $245^\circ\text{C}$ , reducing pressure to less than 3 millimeters of mercury (less than 400 Pa) and agitating for a period of four to eight hours. With polymer molecular weight at the desired level, polymer was extruded through a ribbon or strand die, quenched, and cut into a flake or pellet  
10 size suitable for remelt extrusion or solid state polymerizing. Polymer intrinsic viscosity ("IV") in the range of 0.60 dl/g to 1.00 dl/g was produced by this method.

The polymer made by this process (with  $\text{TiO}_2$ ) was used in Example II-3. The polymers used in Examples II-5, II-6, II-7, II-8, II-9, III-13 and III-14 were  
15 made in substantially the same manner, except that  $\text{TiO}_2$  was not added, and had the same IV. The polymers for Examples II-10 and III-15 were made in the same way, but had a slightly higher IV and did contain  $\text{TiO}_2$ .

#### Polymer Preparation 2

Higher molecular weight polymer (IV  $> 1.00$  dl/g) for Examples II-2, III-  
20 11 and III-12 was produced by solid state polymerizing polymer chip or flake (made in the same way as described above) in a fluidized bed polymerizer. The polymer of Example III-11 included  $\text{TiO}_2$ , whereas the others did not. Crystallized and dried polymer was charged to a fluidized bed reactor continually agitated and purged with dry, inert gas and maintained at a temperature of  $200^\circ\text{C}$   
25 to  $220^\circ\text{C}$  for up to 10 hours to produce polymer with IV up to 1.40.

#### Polymer Preparation 3

Poly(trimethylene terephthalate) polymer for use in Example II-4 was prepared from terephthalic acid and 1,3-propanediol using a two vessel process utilizing an esterification vessel ("reactor") and a polycondensation vessel  
30 ("clave"), both of jacketed, agitated, deep pool design. 428 lb (194 kg) of 1,3-propanediol and 550 lb (250 kg) of terephthalic acid were charged to the reactor. Esterification catalyst (monobutyl tin oxide at a level of 90 ppm Sn (tin)) was added to the reactor to speed the esterification when desired. The reactor slurry was agitated and heated at atmospheric pressure to  $210^\circ\text{C}$  and maintained while  
35 reaction water was removed and the esterification was completed. At this time the temperature was increased to  $235^\circ\text{C}$ , a small amount of 1,3-propanediol was removed and the contents of the reactor were transferred to the clave.

With the transfer of reactor contents, theclave agitator was started and 91 grams of tetraisopropyl titanate was added as a polycondensation catalyst. If titanium dioxide was desired in the polymer, a 20% slurry in 1,3-propanediol was added to theclave in an amount to give 0.3 wt. % in polymer. The process  
5 temperature was increased to 255°C and the pressure was reduced to 1mm Hg (133 Pa). Excess glycol was removed as rapidly as the process would allow. Agitator speed and power consumption were used to track molecular weight build. When the desired melt viscosity and molecular weight were attained, clave pressure was raised to 150 psig (1034 kPa gauge) and clave contents were  
10 extruded to a cutter for pelletization.

TiO<sub>2</sub> was added in the same amount and in the same way as in Polymer Preparation 1.

#### **Polymer of Example II-1**

Batch poly(trimethylene terephthalate) polymer having the properties  
15 described in Table 1 and 0.3 weight % TiO<sub>2</sub> was used for Example II-1.

#### **Example II**

Several samples of poly(trimethylene terephthalate) polymer, prepared as described in Example I, were spun into partially oriented filaments, using a conventional remelt single screw extrusion process and conventional polyester  
20 fiber melt-spinning (S-wrap) process, as illustrated in Figure 1. The spinning conditions and properties for the resulting partially oriented yarns are set forth in Table I. The starting polymers had varying intrinsic viscosities, as indicated in Table I. The polymer was extruded through spinneret orifices having a diameter of about 0.23 mm. The spin block temperature was varied to obtain the polymer  
25 temperatures indicated in Table I. The filamentary streams leaving the spinneret were quenched with air at 21°C and collected into bundles of filaments. Spin finish was applied in the amounts indicated in Table I, and the filaments were interlaced and collected as multi-filament yarn.

Each of the partially oriented yarns spun in this example was suitable as a  
30 very fine denier feed yarn for making drawn yarns according to the present invention, as illustrated in Example IV. Yarn item "II-10" was suitable as a very fine denier direct-use partially oriented yarn in some applications. Such a fine denier partially oriented poly(trimethylene terephthalate) yarn may be woven or knit into end use fabrics without further drawing.

#### **Example III**

35 This example showed the spinning parameters used to spin additional samples of poly(trimethylene terephthalate) polymer into partially oriented filaments. The polymers used in this example were prepared as described in

Example I. The spinning conditions and properties for the resulting partially oriented feed yarns are set forth in Table II. As with the feed yarns from Example II, the partially oriented yarns spun in this example were suitable for making very fine denier drawn yarns. Yarn item "III-15" was also suitable as a very fine denier direct-use partially oriented yarn.

#### Example IV

The partially oriented feed yarns from Example II were drawn at a speed of 400 meters per minute ("mpm") over a heater plate at varying temperatures, with varying draw ratios. The drawing parameters and drawn yarn properties are provided in Table III. As shown in Table III, the yarns of the present invention were drawn such that  $\Delta DR$  is less than ten percent.

Table I

Table I												
Id.	Spinning Conditions				Winding Speed, m/m	Yarn Denier (dtex)	Denier Per Filament (dtex)	Spun Yarn Properties				
	IV	Speed, m/m	Temp, °C	Finish, %				# of Fils.	Ten., g/d (dN/tex)	E <sub>B</sub> , %	Mod, g/d (dN/tex)	DHS, %
II-1	1.04	1829	254	0.60	100	107(119)	1.07(1.19)	2.47(2.18)	128	18.6(16.4)	--	52
II-2	1.2	2743	275	0.50	100	95(106)	0.95(1.06)	2.98(2.63)	83	20.2(17.8)	--	42
II-3	0.88	2743	270	0.50	100	96(107)	0.96(1.07)	2.7(2.38)	98	20.1(17.7)	41	43
II-4	0.88	2746	270	0.50	200	201(223)	1.01(1.11)	2.73(2.41)	91	22.8(20.1)	28	38
II-5	0.88	3200	265	0.60	100	112(124)	1.12(1.24)	2.85(2.52)	82	17.0(15.0)	--	36
II-6	0.88	3200	265	0.60	100	150(167)	1.50(1.67)	2.77(2.44)	81	17.7(15.6)	--	36
II-7	0.88	3200	265	0.60	100	113(126)	1.13(1.26)	2.78(2.45)	83	18.8(16.6)	--	40
II-8	0.88	3200	265	1.00	100	153(170)	1.53(1.70)	2.73(2.41)	75	20.5(18.1)	--	39
II-9	0.88	4115	265	0.60	100	88(98)	0.88(0.98)	3.29(2.90)	60	21.7(19.2)	--	31
II-10	0.92	4115	265	0.50	100	84(93)	0.84(0.93)	3.15(2.78)	63	24.5(21.6)	--	25

Table II

Table II													
Id.	Spinning Conditions					Winding Speed m/m	Yarn Denier (dtex)	Denier Per Filament (dtex)	Spun Yarn Properties			DHS, %	BOS, %
	IV	Speed, m/m	Temp, °C	Finish, %	# of Fils.				Ten., g/d (dN/tex)	E <sub>B</sub> , %	Mod, g/d (dN/tex)		
III-11	1.05	2743	270	0.40	100	2670	96(107)	0.96(1.07)	2.79(2.46)	91	22.7(20.0)	30	37
III-12	1.05	2743	270	0.40	100	2670	95(106)	0.95(1.06)	3.07(2.71)	81	23.4(20.7)	25	29
III-13	0.88	3658	265	1.00	100	3612	137(152)	1.37(1.52)	2.96(2.61)	68	20.7(18.3)	--	30
III-14	0.88	4115	265	1.00	100	4078	123(137)	1.23(1.37)	2.87(2.53)	62	20.1(17.7)	--	17
III-15	0.92	4115	265	0.50	100	4042	78(87)	0.78(0.87)	3.27(2.89)	66	24.4(21.5)	--	27

Table III

Id.	Drawing Conditions		Drawn Yarn Properties						Predicted Draw Ratio	
	Draw Ratio	Heater Plate °C	Yarn Denier (dtex)	Denier Per Filament (dtex)	Tenacity, g/d (uN/tex)	E <sub>B</sub> , %	Modulus, g/d (dN/tex)	DHS, %	BOS, %	Draw Ratio ΔDR, %
IV-1	1.40	130	78(87)	0.78(0.87)	2.98(2.63)	54	21.2(18.7)	--	13.3	1.44 2.86
	1.50		73(81)	0.73(0.82)	3.21(2.83)	43	23.4(20.7)	--	13.9	1.54 2.67
	1.52		73(81)	0.73(0.81)	3.21(2.83)	39	23(20.3)	--	14.0	1.58 3.95
IV-2	1.1	160	88(98)	0.88(0.98)	3.13(2.76)	57	24.5(21.6)	10	7.0	1.15 4.55
	1.2		82(91)	0.82(0.91)	3.59(3.17)	50	23.7(20.9)	13	10.0	1.20 0.00
	1.3		82(91)	0.81(0.90)	3.83(3.38)	38	30(26.5)	16	11.0	1.29 -0.77
	1.4		75(83)	0.75(0.83)	4.06(3.58)	29	28(24.7)	16	13.0	1.38 -1.43
	1.5		67(74)	0.67(0.74)	4.52(3.99)	27	29.3(25.9)	16	13.0	1.39 -7.33
IV-3	1.1	120	88(98)	0.88(0.98)	2.69(2.37)	70	22.4(19.8)	11	8.0	1.15 4.55
	1.2		81(90)	0.81(0.90)	2.71(2.39)	51	23.4(20.7)	15	12.0	1.28 6.67
	1.3		76(84)	0.76(0.84)	3.12(2.75)	45	25.6(22.6)	17	14.0	1.33 2.31
IV-4	1.1	120	186(207)	0.93(1.03)	2.54(2.24)	60	23.1(20.4)	13	10.0	1.18 7.27
	1.2		173(192)	0.86(0.96)	2.84(2.51)	51	25.4(22.4)	16	14.0	1.24 3.33
	1.3		161(179)	0.81(0.90)	2.73(2.41)	36	26.5(23.4)	18	15.0	1.36 4.62
IV-5	1.3	160	85(94)	0.85(0.94)	3.52(3.11)	36	--	--	--	1.30 0.00

Id.	Drawing Conditions		Drawn Yarn Properties						Predicted Draw Ratio	
	Draw Ratio	Heater Plate °C	Yarn Denier (dtex)	Denier Per Filament (dtex)	Tenacity, g/d (dN/tex)	E <sub>B</sub> , %	Modulus, g/d (dN/tex)	DHS, %	BOS, %	Draw Ratio ΔDR, %
IV-6	1.35	160	82(91)	0.82(0.91)	3.69(3.26)	30	--	--	--	1.35 0.00
IV-7	1.3	160	91(101)	0.91(1.01)	3.38(2.98)	34	25.4(22.4)	--	10.6	1.33 2.31
	1.35		87(97)	0.87(0.97)	3.77(3.33)	36	25.7(22.7)	--	11.4	1.31 -2.96
	1.4		84(93)	0.84(0.93)	3.83(3.38)	30	26.3(23.2)	--	11.3	1.37 -2.14
	1.45		81(90)	0.81(0.90)	3.97(3.5)	28	25.8(22.8)	--	11.6	1.38 -4.83
IV-8	1.5	160	109(121)	1.09(1.21)	4.04(3.57)	25	24.1(21.3)	--	12.0	1.36 -9.33
IV-9	1.2	160	71(79)	0.71(0.79)	4.09(3.61)	36	28.4(25.1)	--	10.0	1.16 -3.33
	1.25		72(80)	0.72(0.80)	3.95(3.49)	30	27.7(24.4)	--	10.8	1.21 -3.20
	1.3		75(83)	0.75(0.83)	3.85(3.4)	26	24.3(21.4)	--	10.6	1.24 -4.62
IV-10	1.1	160	74(82)	0.74(0.82)	3.22(2.84)	40	24.6(21.7)	--	8.0	1.15 4.55
	1.2		70(78)	0.70(0.78)	3.48(3.07)	30	25.9(22.9)	--	11.0	1.23 2.50

**WHAT IS CLAIMED IS:**

1. A process for making a drawn yarn comprising:
  - (a) providing partially oriented feed yarn filaments prepared from a polyester polymer having an intrinsic viscosity of at least 0.80 dl/g comprising at least 85 mole % poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units; and
  - (b) drawing the filaments between a set of feed rolls to produce a denier per filament less than about 1.5 and an actual draw ratio within 10 percent of a predicted draw ratio, wherein the predicted draw ratio is determined according to:  $[(\text{elongation to break of the feed yarn}) + 115]/[(\text{elongation to break of the drawn yarn}) + 115]$ .
2. The process of claim 1 further comprising heating the filaments to a temperature greater than the glass transition temperature of the filaments, but less than 200°C, prior to drawing the filaments.
3. The process of claim 1 or 2, further comprising preparing the partially oriented feed yarn filaments by extruding the polyester in a molten state a temperature between about 255°C and 275°C through a spinneret to form filaments.
4. The process of claims 1-3 further comprising interlacing the filaments prior to drawing them.
5. The process of any of the preceding claims, wherein the actual draw ratio is within 5 percent of the predicted draw ratio.
6. The process of claim 5, wherein the actual draw ratio is within 3 percent of the predicted draw ratio.
7. The process of any of the preceding claims, wherein the denier per filament of the drawn yarn is less than 1.0.
8. The process of any of the preceding claims wherein the undrawn filaments have a denier per filament less than about 2.
9. The process wherein the drawing comprises warp drawing or single end drawing and further comprising air jet texturing or false-twisting.
10. A process of preparing a fine denier partially oriented undrawn feed yarn made from a polyester polymer melt-extruded at a spinning temperature between about 255°C and about 275°C, wherein said polymer comprises at least 85 mole % poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units, and wherein said polymer has an intrinsic viscosity of at least 0.80 dl/g, and wherein said partially oriented undrawn fine denier feed yarn has a denier per filament less than about 2.



11. The process of claims 8 or 10 wherein the undrawn filaments have a denier per filament less than about 1.5.
12. The process of claim 11 wherein the undrawn filaments have a denier per filament less than about 1.0.
- 5 13. The process of any of the preceding claims wherein the polymer has an intrinsic viscosity of 0.90 dl/g .
14. The process of any of the preceding claims, wherein the spinning temperature is between 260°C and 270°C.
- 10 15. The process of any of the preceding claims, wherein the polyester is melt-extruded on a spinneret having orifices between about 0.12 to 0.38 mm in diameter.
16. The process of any of the preceding claims, wherein the polymer has an intrinsic viscosity of at least 1.00 dl/g.
17. A yarn prepared by the process of any of the preceding claims.
- 15 18. A drawn yarn prepared from a polyester polymer having an intrinsic viscosity of at least 0.80 dl/g comprising at least 85 mole % poly(trimethylene terephthalate) wherein at least 85 mole % of repeating units consist of trimethylene units, wherein the drawn yarn has a denier per filament less than about 1.0.

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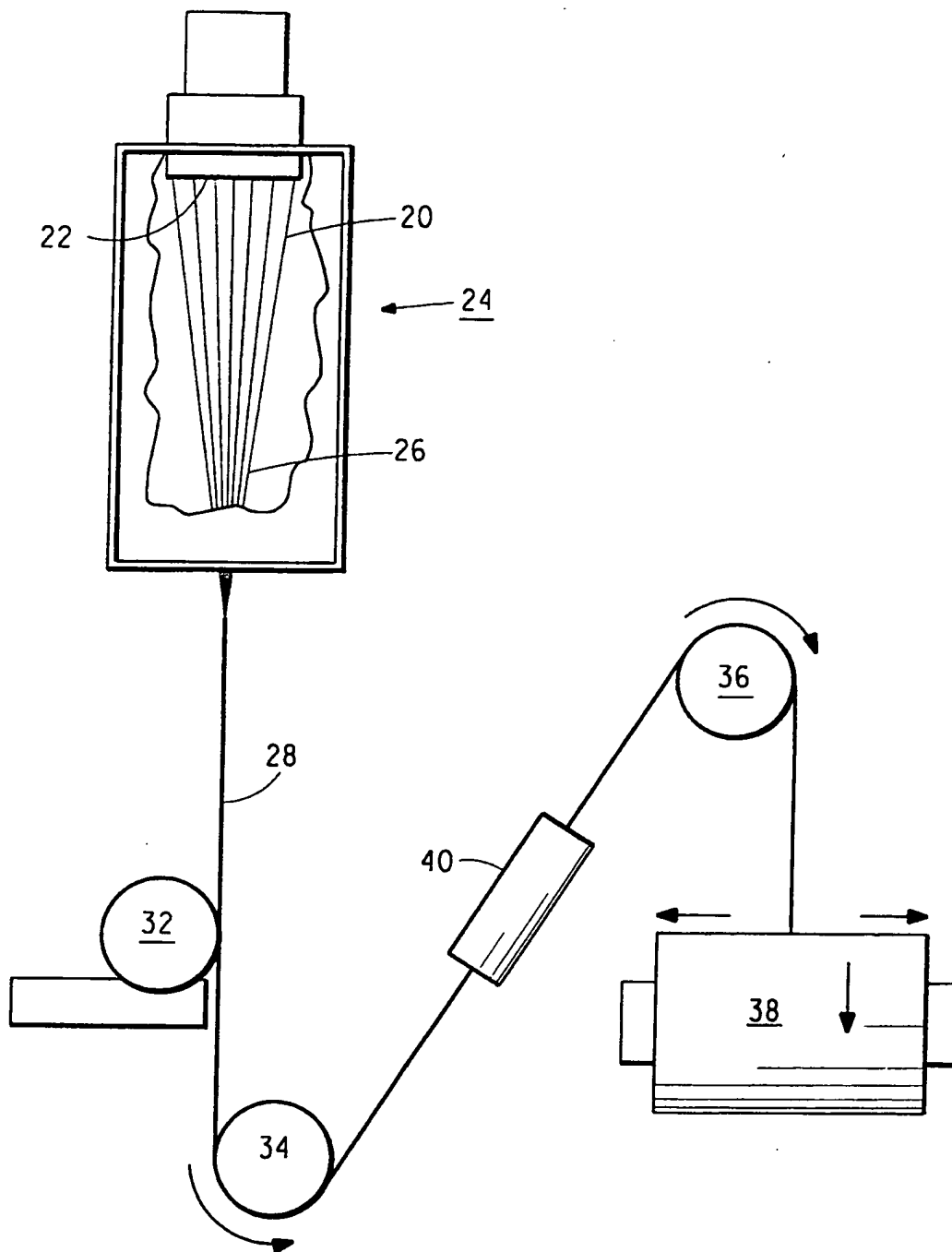


FIG 1

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 01/06567

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 D01F6/62

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 D01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	EP 1 033 422 A (ASAHI CHEMICAL IND) 6 September 2000 (2000-09-06) the whole document	1-8, 13-18
X	example COMP11 & WO 99 27168 A 3 June 1999 (1999-06-03) ----	10-12
P, X	EP 1 052 325 A (ASAHI CHEMICAL IND) 15 November 2000 (2000-11-15) the whole document & WO 99 39041 A 5 August 1999 (1999-08-05) -----	1-9, 13-18

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

20 July 2001

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06/08/2001

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# INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

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